Kentucky Method 64-253-03
Revised 1/7/03
Supersedes 64-253-00
Dated 1/27/00

CHEMICAL ANALYSIS ~~TESTING~~ OF FLY ASH

1. SCOPE: This method is a modification of ASTM C-114, as referenced in ASTM C-311, for the chemical testing of fly ash by x-ray spectroscopy.

2. APPARATUS – MATERIALS:

   2.1. Philips MagiX PRO Wavelength Dispersive x-ray fluorescence spectrometer.

   2.2. SuperQ software

   2.3. Philips Perl'x 3 fused bead machine

   2.4. Platinum dish and crucible set

   2.5. Spex 8000 mixer/mill

   2.6. Lithium Bromide (LiBr): 10% solution non-wetting agent

   2.7. 67% Lithium Tetraborate (Li2B4O7) : 33% Lithium Metaborate (LiBO2) flux

3. PROCEDURE:

   3.1. Weigh approximately 2 grams of fly ash sample into a small beaker.

   3.2. Dry the sample to a constant weight in an oven at 110°C.

   3.3. Calculate the moisture content using the following formula:

   MC = (A/B) x 100

   where: A = weight of sample after drying;
   B = weight of original sample.

   3.4. Prepare porcelain crucibles by igniting at 750°C to a constant weight. Cool and store crucibles in a dessicator to avoid absorption of moisture

   3.5. Weigh 1.0 gram of fly ash sample from the moisture analysis accurately to 0.0001 grams into a prepared porcelain crucible. Ignite sample to a constant weight in a muffle furnace at 750°C and cool in a dessicator.

3.6. Calculate the loss on ignition (LOI) using the following formula:

LOI = (A/B) x 100

where: A = weight of sample after ignition;
B = weight of moisture free sample.

3.7. Place at least 1 gram of fly ash sample from LOI in the spex 8000 mixer/mill and grind until a fine powder is obtained and/or sample is completely uniform. This takes approximately 30 seconds.

3.8. Weigh accurately to 0.0001 grams 6.0 grams of 67:33 flux directly into platinum crucible. Then weigh accurately to 0.0001 grams 0.6 grams of fly ash sample from the spex 8000 mixer/mill directly into the platinum crucible. Add 3 drops of LiBr solution. Place the platinum crucible and dish in the Perl'x 3 machine and select to run program 7* for all types of fly ash. This takes approximately 15 minutes. Program run is dependent on sample type.

3.9. Enter names of samples and LOI information in measure sample screen on the measure and analyze program.

3.10. Place sample in a 27mm steel cup. Then place in x-ray instrument and prepare to run fly ash application on the measure sample screen. Click on measure at the bottom of the screen. This may take a few minutes. The application chosen is dependent upon the sample type.

4. QUANTIFICATION: Program quantifies data by using a least squares program. Similar samples with known chemical makeups are used as standards in the quantification technique. As many standards as possible are used for best quantification. The results are reported as oxides in weight percents.

5. REPORT:

5.1. % Moisture Content

5.2. % LOI

5.3. % SO3

5.4. % Al2O3

5.5. % Fe2O3

5.6. % R value: for class C fly ashes only. [(CaO – 5)/ Fe2O3]

5.7. % MgO

5.8. % Na2O

5.9. % K2O

5.10. % CaO

5.11. % SiO2

* NOTE: Program 7 for fly ash includes: One oxidation for 4 minutes, temperature 1100°C, generator power 77, agitation angle 30, and agitation speed 15. One fusion for 5 minutes, temperature 1100°C, generator power 77, agitation angle 60, and agitation speed 20. Then a pause before casting for 10 seconds at a temperature of 1100°C. Casting time 10 seconds, temperature 1100°C, casting angle 123, casting speed 10, and time for solidification 2 minutes. Lastly there is natural air cooling for 4 minutes and forced air cooling for 2 minutes at a flow rate of 40. The setting of the dish height dial is 12/40 this depends on the size of the platinum dish being used.

~~1. SCOPE: This method is a modification of ASTM C-114, as referenced in ASTM C-311, for the chemical testing of fly ash by X-ray spectroscopy.~~

~~2. APPARATUS and REAGENTS:~~

~~2.1. Kevex: Analyst 771 Energy Dispersive X-Ray Fluorescence Spectrometer.~~

~~2.2. WinXRF software~~

~~2.3. Carver hydraulic press~~

~~2.4. Hardened Steel 31mm die set~~

~~2.5. Spex 8000 mixer/mill~~

~~2.6. Polyvinyl Alcohol (PVA)~~

~~3. PROCEDURE:~~

~~3.1. Weigh 6 grams of fly ash sample accurately to 0.0001 grams. Weigh accurately to 0.0001 grams 1.0 grams of polyvinyl alcohol. Calculate the added/sample ratio. Calculation:~~

~~Weight polyvinyl alcohol/weight of fly ash = Added/Sample Ratio~~

~~3.2. Place both components in the Spec 8000 mixer/mill, and grind until a fine power is obtained and/or sample is completely uniform. This takes approximately 30 seconds.~~

~~3.3. Place the homogeneous sample in the die. Press at 33,000 lbs and allow pressure to release very slowly. This make take up to 45 minutes.~~

~~3.4. Enter names of samples and Added/Sample ratio information by running NAMES program in the cement library.~~

3.5. Place sample in machine and prepare to run program FLYASHC or FLYASHF. Program run is dependent on sample type.

3.6. Condition Codes: Program will run through a series of condition codes. The condition code acquires a spectrum, subtracts escape peaks, models and subtracts the background using windows described in Table 3.2, and deconvolutes for certain elements using deconvolution files described in Table 3.3 The peak intensities are saved.

3.6.1. Code 1: Deconvolutes spectra for Al, Si, and S.

3.6.2. Code 2: Deconvolutes spectra for Fe and Ca.

3.6.3. Condition Code 3: Deconvolutes spectra for Mg.

3.7. Quantification: Program quantifies data by using a Least Squares Program. Similar samples with known chemical makeups are used as standards in quantification technique. As many standards as possible are used for the best quantification. The results are reported as oxides.

TABLE 3.1 CONDITIONS

Condition Code	kV	mA	Secondary Target	Acquire Time (Sec)	Spectra Size
1	3.5	.28	Direct	700	1024
2	19.0	1.50	GE	350	2048
3	20.0	3.27	AL	700	1024

All conditions are set: Gain = 40, Shaping Index = 32, eV/Channel = 10

TABLE 3.2 BACKGROUND WINDOWS

Fly ash F

Condition 1		Condition 2		Condition 3	
Low	High	Low	High	Low	High
.09	.95	0.14	1.33	0.06	0.94
1.39	1.60	1.92	3.15	1.13	1.38
1.61	1.87	3.58	3.84	1.91	2.24
1.88	2.15	4.22	4.31	2.39	2.89
2.17	2.44	5.10	5.19	3.87	3.90
		5.73	6.57	4.11	4.34
		7.28	7.49	4.74	4.77
		7.63	7.85	5.08	5.28
		8.24	8.49	5.54	5.74
				8.23	8.53
				8.57	9.53

Fly ash C

Condition 1		Condition 2		Condition 3	
Low	High	Low	High	Low	High
0.06	1.10	0.20	1.38	0.08	0.84
2.56	8.42	2.42	3.12	2.48	3.14
		4.24	4.30	4.18	4.30
		5.02	5.72	5.10	6.10
		7.62	7.82	7.26	8.48
		8.28	8.46	9.10	9.44
		8.84	9.26	9.76	23.58
		10.10	10.36		

~~TABLE 3.3 DECONVOLUTION~~

~~Condition 1~~	~~Condition 2~~	~~Condition 3~~
~~Al 1.32 - 1.57~~	~~Ca 3.5 – 3.84~~	~~Mg 1.12 – 1.32~~
~~Si 1.59 - 1.91~~	~~Fe 6.09 – 6.84~~	
~~S 2.17 - 2.44~~		

APPROVED _____
Director
DIVISION OF MATERIALS

DATE _____1/7/03_____

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